

UDC 666.762.852:666.3.032.4:436.421.48

MOLDING SILICON CARBIDE ARTICLES BY FREEZING AQUEOUS SLIP

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Translated from *Steklo i Keramika*, No. 8, pp. 10–11, August, 2001.

Molding silicon carbide articles by freezing aqueous slip is considered. The freezing conditions are determined. The slip composition is selected. Firing in a nitrogen-hydrogen medium at 1850°C made it possible to obtain samples whose properties are typical of this material.

Silicon carbide articles are produced by dry pressing, plastic molding, and ramming [1], and articles of a more complex shape are produced by slip casting. In the course of aqueous casting into porous (gypsum) molds, it is essential to control such process parameters as the mixture setting rate and shrinkage in curing needed to remove the cast article from the mold. In casting silicon carbide articles using aqueous slip, the molds get quickly worn [2]. The main drawback of the hot slip-casting method is the use of paraffin and similar materials as a temporary technological binder. The use of paraffin involves such power- and labor-consuming operations as material preparation and burn-out of the binder [3]. Moreover, burning of the binder represents an environmental risk.

The purpose of the present study was to develop a method for molding silicon carbide articles of complicated shapes that would eliminate the above drawbacks.

Such a method could be freezing aqueous slip in a metal mold. The slip consisted of powdered silicon carbide and carbon (soot). The introduction of carbon makes it possible to produce articles based on self-bonded silicon carbide in the course of silication of the SiC–C mixture.

In the course of study, it was necessary to select the freezing conditions, determine the vacuum drying conditions and the possibility of replacing it with air drying, and evaluate the properties of the samples obtained.

Several compositions were selected for the solid phase of slip (Table 1).

The slip was prepared using 5% aqueous solution of polyvinyl alcohol, which was selected for the following reasons: PVA improves the wettability of the soot surface, i.e., acts as a surfactant; PVA is an important component of slip, since in freezing and defrosting it is restructured with the formation of gel; therefore, a cast article does not get deformed when taken out of the mold at a temperature above the freez-

ing point; PVA imparts additional strength to the articles being taken out of the mold and transported onwards (U.S. patent No. 5861115) [4, 5].

The materials used were PVA of grade Mowiol 56-98 with molar weight 195,000 g/mole produced by Clariant Co., silicon carbide (green) M10 and M40, technical carbon P803 with a particle size of less than 1 μm (petrographic analysis data) and pycnometric density 1.85 g/cm³. PVA was introduced in the amount of 2% of dry material weight.

Soot was mixed with the PVA solution and water. Then silicon carbide powdered mixture was introduced in the prepared suspension, and the slip was mixed for 1 h. The moisture of the resulting slip was 42%. Since foaming was observed in the slip mixing, octyl alcohol was added as a foam quencher.

The samples were molded in the shape of crucibles 22 mm high with an outer diameter of 24 mm and a wall thickness of 3 mm.

The slip was poured through the pouring gate into a metal mold, which was placed in a freezer chamber. The molds can be either metal or polymer. Freezing was carried out at a temperature of –32°C. After exposure the mold was taken out of the freezer and left in air. The optimum freezing duration was selected within the interval from 2 to 40 min. The sample froze 6 min after the start of freezing; however, it did not acquire sufficient strength for removing it from the mold and was deformed.

The samples held at a minus temperature for more than 15 min had sufficient strength for removing them from the

TABLE 1

Composition	Weight content %		Ratio SiC _{M10} : SiC _{M40}
	C	SiC	
1	30	70	40 : 60
2	40	60	40 : 60
3	50	50	40 : 60

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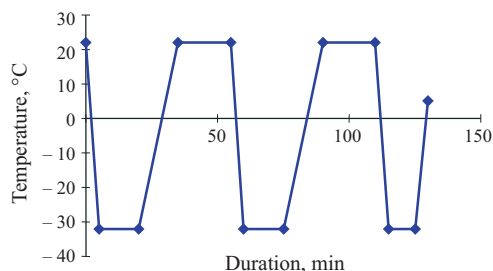


Fig. 1. Freezing conditions.

mold. Therefore, the optimum duration for a single freezing cycle was 15 min.

In a single freezing cycle, the articles removed from the mold and exposed at room temperature with time became deformed as a consequence of ice melting, and the samples cracked. Therefore, sublimation drying was used, which lasted 25 min.

Patent No. 5861115 (U.S.) established that in one-time freezing and defrosting, as well as in using high-molecular PVA, the cast article preserves its configuration after its extraction from the mold and a protracted exposure in air. Therefore, freezing was carried out in several modes, and finally the mode with the minimum number and minimum duration of cycles was selected for samples of this particular size: freezing duration 20 min, defrosting 35 min, and number of cycles equal to 2 (Fig. 1).

The specified freezing mode makes it possible to obtain a sufficiently strong article and eliminate the vacuum drying stage. The shrinkage of samples in air drying was 4%.

The samples were fired in a vacuum furnace in a nitrogen-hydrogen gas mixture at temperature of 1850°C. The shrinkage of samples of compositions 1 and 2 after firing was 0.1%. The samples of composition 3 became destroyed in firing.

The samples obtained as a result of the present study had properties which are typical of the given material.

Thus, the method for molding samples made of silicon carbide and soot by freezing aqueous slip has been evaluated. It is recommended to use a 5% PVA solution as a temporary technological binder and a surfactant. The use of a high-molecular modification of PVA makes it possible to eliminate the sublimation drying of the article.

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